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Short communication

Thermoregulated ionic liquid-coordinating ruthenium complexes for asymmetric hydrogenation of aromatic ketones

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ABSTRACT

This work presented the synthesis and characterization of new ionic liquid-coordinating ruthenium complexes. The resulting ruthenium complexes exhibited not only excellent thermoregulated phase-separation behavior but also highly catalytic activity and enantioselectivity for the asymmetric hydrogenation with molecular hydrogen. The thermoregulated ionic liquid catalyst was highly resistant to leaching and was recycled consecutively for six times without significant loss of catalytic activity and enantioselectivity. The presence of Ru—H species revealed that NH and a Ru—H unit, involved in the hydride transfer process, were of great importance in the present catalytic system.

1. Introduction

Asymmetric hydrogenation of prochiral ketones is one of the most remarkable methods for the industrial production of chiral alcohols [1,2]. Chiral alcohols are significant intermediates for synthesizing chiral drugs and fine chemicals, so that the synthesis of chiral alcohols is indispensable nowadays. One of the most effective methods is the asymmetric hydrogenation of prochiral compounds using a transition-metal-complex as the chiral catalyst. With regard to the catalyst-designing, introducing chiral ligands is distinctly important for synthesis. Among these methodologies, asymmetric hydrogenation of simple ketones catalyzed by the homogeneous chiral Ru(II) complexes discovered by Noyori et al. showed excellent characteristics of activity and enantioselectivity [3,4].

Discovering green approaches towards synthesis of chiral compounds is of great concern. However, homogeneous catalysts often have problems of catalyst separation and reuse. Thus, designing easily separable catalysts is a popular and interesting research area. The immobilization of homogeneous catalysts on insoluble supports may solve this problem, but it is difficult to maintain the same high level of catalytic performance as the original homogeneous counterparts, because the decrease of the catalyst dispersion in the microscopic reaction environment could increase the mass transfer resistance between the substrate and the active sites.

Up to now, the methods of fluorination [5], silanization [6] and magnetic adsorption [7] have been reported to create “smart” catalytic systems. Recently, our group has reported several catalytic systems with temperature-controlled [8–10] or pH-sensitive [11] phase separation in solvents such as ethyl acetate, isopropanol or aqueous phase. These catalyst systems provided not only the advantages of convenient phase-separation like heterogeneous catalysts, but also the catalytic activity as high as homogeneous counterparts. Among these methodologies, the thermoregulated phase-separation system afforded an alternative approach to designing high-performance catalysts, and showed the advantages of high reusability, mild reaction conditions and even the potential suitability with low catalyst loading [12].

For hydrogenation reaction, the normal hydrogen source is H_2 gas itself, which is typically available commercially within the storage medium of a pressurized cylinder. The hydrogenation process often uses > 1 atm of H_2, in order to achieve excellent conversions. As compared with transfer hydrogenation, using H_2 gas as hydrogen source is clearly greener, and H_2 can be separated more easily from the reaction mixture [13–15].

In the previous research, we have developed thermoregulated ionic liquid (IL) catalysts for selective hydrogenation of C=C bonds [16], as well as asymmetric transfer hydrogenation of aromatic ketones [17]. Herein, we report on the synthesis of new thermoregulated IL catalysts consisting of poly(ethylene glycol) (PEG) functionalized imidazolium as a cation moiety and ruthenium complexes coordinated with diphosphine and sulfonated diamine ligands as an anion moiety. The IL catalysts exhibits strong thermoregulated behavior and provided an alternative approach for highly efficient asymmetric hydrogenation of

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various ketones using H_2 gas as a green hydrogen source. Additionally, the catalytic reaction can be performed under mild reaction conditions, bearing a satisfying reusability without obvious loss of catalytic performance.

1.1. Experimental section

The PEG chain-functionalized 1-alkyl-2-methyl-imidazolium dichlorides ([PEG-1000-C_nMIM]Cl_2) and the sulfonated (*S*,*S*)-1,2-diphenyl-ethylenediamine ((*S*,*S*)-DPENDS, sodium salt) were prepared according to the our previously reported procedure [17], and then the anion moiety (Ru-BINAP-DPENDS) was prepared through the reaction between [*p*-(cymene)$RuCl_2$]$_2$ and (*S*)-(−)-2,2'-Bis (diphenylphosphino)-1,1′-binaphthyl ((*S*)-BINAP), followed by reacting with (*S*,*S*)-DPENDS. Thus the IL-regulating ruthenium complex ([PEG-1000-C_nMIM][Ru-BINAP-DPENDS]) was obtained by ion exchange of [PEG-1000-C_nMIM]Cl_2 and Ru-BINAP-DPENDS. All synthetic procedures and characterization of the IL-regulating ruthenium complexes have been given in the supporting information (Scheme S1-S5).

2. Results and discussion

2.1. Catalyst characterization

It has been demonstrated that PEG-functionalized 1-alkyl-2-methyl-imidazolium chloride ([PEG-m-C_nMIM]Cl_2) performs a clear thermoregulated feature [17,18]. In this work, the thermoregulated IL-coordinating ruthenium complexes consisting of the cation moiety [PEG-1000-C_nMIM] and the anion moiety Ru-BINAP-DPENDS have been synthesized, respectively (Scheme S1-S3).

Next, the anion moiety Ru-BINAP-DPENDS was characterized in detail by means of ^{1}H NMR, ^{31}P NMR, FT-IR, ICP-AES and Elemental Analysis. The ^{1}H NMR characterization has been given in the supporting information, where the double peaks at 3.21 could be characterized as the protons from NH_2-*CH*-, and the multi-peaks at 6.51–7.99 came from the protons of aromatic rings. The FT-IR spectrum of the anion moiety Ru-BINAP-DPENDS was shown in Fig. 1b(Left), which gave bands at approximately 1035 cm^{-1}, 1636 cm^{-1}, 1192 cm^{-1} and 1434 cm^{-1} (Fig. 1b), corresponding to the vibration of C—N bond, the aromatic benzene rings (C=C stretching), O=S=O bonds and N—H bond, respectively. Notably, the C—N band in Ru(II) complex anion (1035 cm^{-1}) shifted to a lower wavenumber in comparison to the same band in (*S*,*S*)-DPENDS (1050 cm^{-1}) (Fig. 1a *vs* 1b), suggesting that Ru was successfully coordinated with diamine ligand. All data above confirmed the formation of the proposed Ru(II) complexes with the diamine and diphosphine ligands. In addition, ^{31}PNMR spectrum of Ru-BINAP-DPENDS gave a resonance signal around 58.7 (Fig. 1e, Right), which was slightly larger than the unsulfonated analogue from the literature [19], which could be attributed to the effect of the electron-withdraw group (SO_3^-) tethered to the DPEN ligand.

Besides, the IL cation [PEG-1000-C_nMIM]Cl_2 was conveniently prepared and characterized by ^{1}H NMR and Elemental Analysis (Scheme S3 and S4), which was in well agreement with our previously reported work [9]. At last, the IL catalyst [PEG-1000-C_nMIM][Ru-BINAP-DPENDS] was obtained through simple ion exchange. The FT-IR spectra of the IL catalysts showed the characteristics of both the IL cation and anionic moiety. As shown in Fig. 1c, the bands at approximately 620 cm^{-1} and 1110 cm^{-1} could be characterized as the C—O and C=N bond from the part of IL cation, while the bands at about 1035 cm^{-1}, 1636 cm^{-1}, and 1192 cm^{-1} were characteristics of C—N bond, the aromatic benzene ring (C=C stretching) and O=S=O bonds from the moiety of anion, respectively. Furthermore, the UV/vis spectra of (*S*,*S*)-DPENDS, [(*p*-cymene)$RuCl_2$]$_2$ and [PEG-1000-C_{12}MIM][Ru-BINAP-DPENDS] are shown in Fig. S1. It was observed that the absorbance at 240–280 nm can be assigned to adsorption of aromatic rings (Fig. S1a-c) and the absorbance at 415 nm was attributed to Cl p → Ru d transition (Fig. S1b). However, the band at 415 nm was moved to 560 nm after [(*p*-cymene)$RuCl_2$]$_2$ were reacted with diamine and diphosphine ligands, indicating that Ru(II) complex coordinating with diphosphine and diamine ligands was formed.

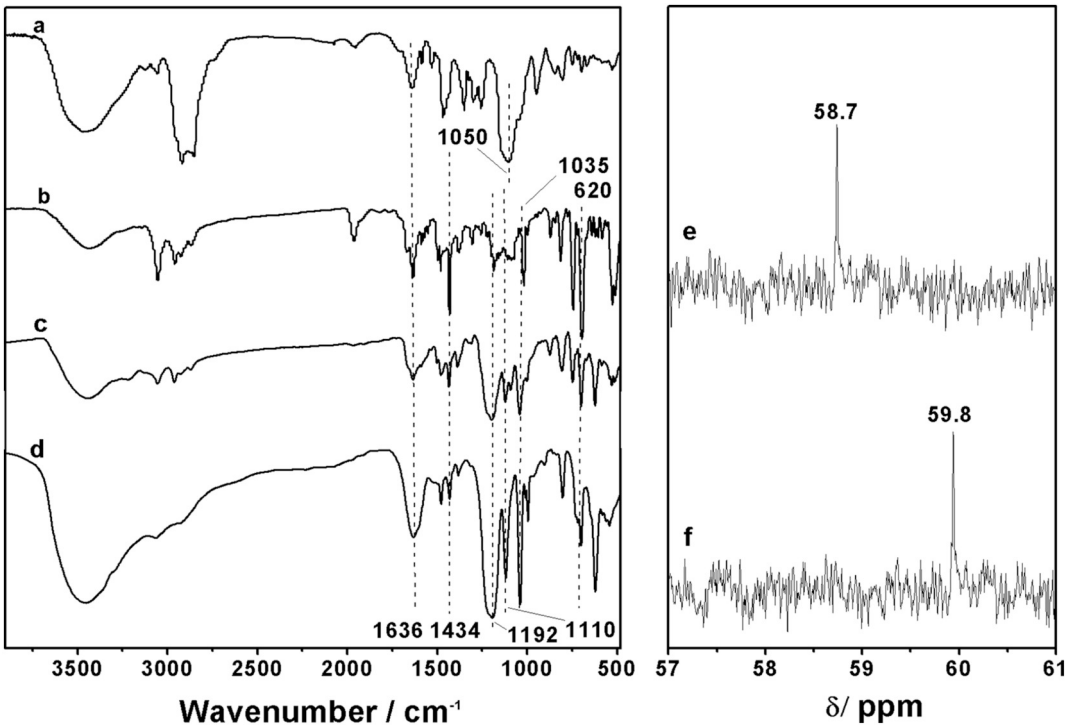


Fig. 1. Left: FT-IR Spectra of a) (*S*,*S*)-DPENDS; b) Ru-BINAP-DPENDS; c) [PEG-1000-C_{12}MIM][Ru-BINAP-DPENDS]; d) the spent [PEG-1000-C_{12}MIM][Ru-BINAP-DPENDS] catalyst. Right: ^{31}P NMR spectra of e) Ru-BINAP-DPENDS in D_2O; f) [PEG-1000-C_{12}MIM][Ru-BINAP-DPENDS] in $CDCl_3$.

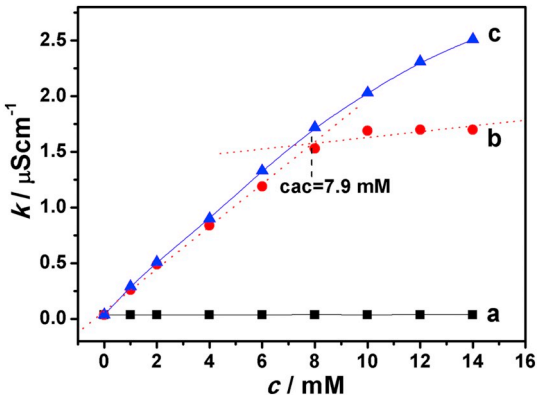


Fig. 2. The conductivity as function of the concentration of the IL [PEG-1000-C_{12}MIM][Ru-BINAP-DPENDS] in isopropanol at a) 0 °C, b) 30 °C and c) 40 °C.

2.2. Thermoregulated phase-separation

At first, the properties of the thermoregulated IL catalysts have been examined. As shown in Fig. S2a, before the reaction, the catalyst sank to the bottom. However. After temperature arose to 40 °C in the presence of base promoter and acetophenone, the reaction mixture became homogeneous (Fig. S2b). As the reaction temperature declined, the catalyst gradually precipitated from the reaction system and sank to the bottom or adhered to inner wall of the flask again (Fig. S2c).

The conductivity has been proved a powerful tool to determine the critical associating concentration (cac) of polymer surfactants [20], which was highly related with the aggregation state of the surfactants. The aggregation of the IL catalyst [PEG-1000-C_nMIM][Ru-BINAP-DPENDS] in isopropanol was investigated by conductivity measurement. As shown in Fig. 2, the conductivity was measured as a function of the concentration of the IL catalyst. It was found that the conductivity of the IL catalyst in isopropanol was close to that of isopropanol at 0 °C (0.06 k/μS·cm^{-1}) (Fig. 2a), demonstrating that the IL was hardly dissolved in isopropanol, which was consistent with visual observation. However, the conductivity increased significantly with the catalyst concentration at 30 °C, and then the increment slowed down as the concentration became higher, indicating that the aggregation indeed occurred, and the cac of IL catalyst was found around 7.9 mM by linear fitting (Fig. 2b). Moreover, as the temperature increased to 40 °C (Fig. 2c), the conductivity increased monotonously with the concentration of IL, which reflected that the aggregation did not almost happen and the IL catalyst possibly existed almost as a molecular form (loose ion pairs) in isopropanol [21]. Additionally, the conductivity was strongly dependent on temperature at the constant concentration (Fig. S3). As the temperature decreased, the conductivity decreased as well, and some of the catalysts were observed to precipitate from solution at around 20 °C. Furthermore, the complete phase separation of catalyst occurred and meantime the conductivity of solution was 0.06 k/μS·cm^{-1} at 0 °C, which was close to the conductivity of isopropanol. These properties were closely relevant to Upper Critical Solution Temperature (UCST) behavior of the IL, which might be attributed to hydrogen bonding or other possible interactions between IL and isopropanol. As the temperature decreased, the intermolecular interaction between IL and solvent was weakened, while the intramolecular interaction in ILs was strengthened, which led to a complete phase separation [22].

2.3. Catalytic asymmetric hydrogenation

In this work, the as-synthesized different IL-coordinating ruthenium complexes have been applied to the asymmetric hydrogenation, and acetophenone was chosen as a model substrate (Table 1). It can be found that the asymmetric hydrogenation hardly happened in the absence of catalyst or hydrogen, confirming that both the IL catalyst and molecular hydrogen were essential for this reaction (entries 1 and 2, Table 1). Although two IL-regulating Ru(II) complexes catalysts [PEG-1000-C_8MIM][Ru-BINAP-DPENDS] and [PEG-1000-C_{12}MIM][Ru-BINAP-DPENDS] exhibited excellent activity and enantioselectivity for hydrogenation of acetophenone, the [PEG-1000-C_8MIM][Ru-BINAP-DPENDS] with shorter carbon chain could not be completely separated from the catalytic system even the temperature decreased to 0 °C (entries 3 *vs* 4, Table 1). This revealed that the thermoregulated separation behavior was dependent strongly on the hydrophobicity of cation moiety, and the appropriate hydrogen bonding or hydrophobic interactions between IL and solvent was very vital to offer the thermomorphic behavior [23,24]. The simple Ru(II) complex salt Ru-BINAP-DPENDS afforded lower catalytic activity and enantioselectivity than that of [PEG-1000-C_{12}MIM][Ru-BINAP-DPENDS] (entries 4 *vs* 6, Table 1) because of its poor miscibility with isopropanol.

The diphosphine ligand (*S*)-BINAP is very crucial to achieve high enantioselectivity. When the Ru sites were coordinated with achiral diphosphine ligand (1,3-Bis(diphenylphosphino)propane) (DPPP) and chiral diamine ligand (*S*,*S*)-DPENDS, the resulting catalyst [PEG-1000-C_{12}MIM][Ru-DPPP-DPENDS] exhibited lower activity and enantioselectivity with respect to the catalyst [PEG-1000-C_{12}MIM][Ru-BINAP-DPENDS] (entries 4 *vs* 5, Table 1) under the same reaction conditions, although the catalyst indeed exhibited temperature-regulated separation behavior. The moiety of achiral diphosphine (DPPP) ligand accounts for low enantioselectivity, although chiral ligand (*S*,*S*)-DPENDS still could exert an impact on asymmetric induction in Ru(II) complex catalyst [25]. Thus, the diphosphine ligand (*S*)-BINAP played a vital role in not only offering chirality but also enhancing enantioselectivity in present IL catalytic systems.

Base promoter is very crucial in asymmetric hydrogenation [26], and then the effect of the base promoter on reaction has been examined over [PEG-1000-C_{12}MIM][Ru-BINAP-DPENDS] catalyst. As shown in Table S1, the conversion and enantioselectivity of asymmetric hydrogenation of acetophenone decreased as the basicity weakened ($(CH_3)_3COK$ > KOH > K_2CO_3) under the same conditions (entries 2, 4 and 5, Table S1). Next, the dosage of base promoter was also examined, which indicated that once $n_{(CH3)3COK}$:$n_{catalyst}$ reached up to 40 (entries 1–3, Table S1), the reaction could happen smoothly. Moreover, the molar ratio of substrate to catalyst ($n_{substrate}$: $n_{catalyst}$) also affected the catalytic activity and enantioselectivity significantly. As shown in Table S2, the conversion of acetophenone decreased distinctly with increasing $n_{substrate}$: $n_{catalyst}$ due to catalyst dilution. The present IL catalyst could afford a TON value as high as *ca.* 3024 within 4 h reaction time (entry 2, Table S2).

With the optimal usages of base promoter and catalyst, the effects of the reaction time, temperature and H_2 gas pressure on the catalytic performance were investigated accordingly. The conversion of the acetophenone was improved to some extent with the increase of the reaction time, but the enantioselectivity declined for a longer reaction time (Fig. S4a), which could result from racemization reaction of the chiral product *R*-1-phenylethanol. Additionally, the reaction temperature had a significant effect. As shown in Fig. S4b, the conversion of acetophenone increased obviously, but the e.e. value of the product decreased slightly in the range of temperature from 20 °C to 40 °C. However, as temperature was above 40 °C, all acetophenone was almost converted to *R*-1-phenylethanol, but the e.e. value reduced to a certain extent, indicating that the high e.e. value could not survive at high temperature. Besides, it was observed that as the H_2 pressure increased from 0.1 MPa to 4.0 MPa (Fig. S4c), the conversion of acetophenone increased from 35% to 99%, and e.e. value remained high above 92%, which revealed that the present IL catalyst can afford high hydrogenation activity, and meantime retained excellent enantioselectivity under high H_2 pressure.

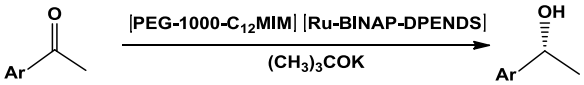
The applicability of catalyst to various substrates is one of the essential standards to evaluate a new catalyst. The asymmetric

Table 1
Catalytic activity, enantioselectivity and thermoregulated performance of the different catalysts for asymmetric hydrogenation of acetophenone.[a]

Entries	Catalysts	Con. (%)[b]	e.e. (%)[c]	Thermoregulated phase separation
1	none	–	–	–
2[d]	[PEG-1000-C_{12}MIM][Ru-BINAP-DPENDS][c]	< 1	–	Yes
3	[PEG-1000-C_8MIM][Ru-BINAP-DPENDS]	92	94	No
4	[PEG-1000-C_{12}MIM][Ru-BINAP-DPENDS]	99	97	Yes
5	[PEG-1000-C_{12}MIM][Ru-DPPP-DPENDS]	52	43	Yes
6	Ru-BINAP-DPENDS	76	92	No

[a] Reaction conditions: 40 °C, 4 h, 3.0 MPa H_2, 0.01 mmol catalyst; 2 mL isopropanol; $n_{catalys}$:$n_{(CH3)3COK}$:$n_{substrate}$ = 1:40:2400, where $n_{catalys}$, $n_{(CH3)3COK}$ and $n_{substrate}$ represented the corresponding moles, respectively.
[b] The selectivity towards *R*-1-phenylethanol was normally over 99%.
[c] GC analysis was performed with a β-DEX™ 120 capillary column (30 m × 0.25 mm, 0.25 μm film) and dodecane as internal standard.
[d] The reaction was carried out in 3.0 MPa N_2.

Table 2
Asymmetric hydrogenation of various ketones catalyzed by [PEG-1000-C_{12}MIM][Ru-BINAP-DPENDS] in isopropanol[a].



Entries	Ar	Time (h)	Con. (%)[b]	e.e. (%)[c]
1	C_6H_5-	4	99	97
2	$C_6H_5C_2H_4$-	4	98	92
3	4-BrC_6H_4-	4	98	99
4	4-ClC_6H_4-	4	97	95
5	2-ClC_6H_5-	4	95	93
6	3-ClC_6H_5-	4	98	93
7	4-$NO_2C_6H_4$-	4	92	96
8	4-MeOC_6H_4-	4	96	93
9	2-MeC_6H_5-	4	64	89
10	4-MeC_6H_4-	6	95	99
11	4-IsobutylC_6H_4-	6	69	98
12	4-Furyl-	4	89	99
13	2-Naphthyl-	4	95	92

[a] Reaction conditions: 40 °C, 3.0 MPa H_2, 0.01 mmol catalyst; 2 mL isopropanol; $n_{catalys}$:$n_{(CH3)3COK}$:$n_{substrate}$ = 1:40:2400.
[b] The selectivity towards the corresponding secondary alcohols was normally over 99%.
[c] GC analysis was performed with a β-DEX™ 120 capillary column (30 m × 0.25 mm, 0.25 μm film) and dodecane as internal standard.

hydrogenation reactions of a wide range of ketones, such as heterocyclic aryl ketones, naphthylacetones and other derivatives with different substituents were carried out as shown in Table 2. Under the mild reaction conditions, the aromatic ketones with electron-withdrawing substituents were basically hydrogenated to produce corresponding chiral products (entries 2–7, Table 2). For the aromatic ketones with electron-donating substituents, the hydrogenation activity of the catalysts decreased comparably (entries 8–11, Table 2), but the enantioselectivity of the products still kept above 85%, which indicated that the catalyst system was influenced by the electron effects. An electron-donating group, such as a methyl group or a methoxy group, caused an increase in the electron density on the benzene ring and the carbonyl group, so that the carbon cation on the carbonyl group became less susceptible to protons and reduced the possibility to be hydrogenated. Notably, no products related to dehalogenative hydrogenation nor the reduction of nitro group have been detected with the present Ru(II) complex catalyst during the course the reaction (entries 3–6, 8, Table 2). As for heterocyclic aromatic substrates, such as 2-acetylfuran, the catalysts exhibited high activity and enantioselectivity as well (entry 12, Table 2). Moreover, naphthylacetone also gave efficient conversion and enantioselectivity and no by-products were detected in this catalytic system (entry 13, Table 2). In a word, the catalyst showed an excellent applicability on different substrates. The GC trace analysis of asymmetric hydrogenation of various aromatic ketones were afforded in Fig. S10-S25 in Supporting Information.

The recyclability of the catalyst [PEG-1000-C_{12}MIM][Ru-BINAP-DPENDS] was examined by choosing the acetophenone as a model substrate in isopropanol. With simple decantation of the products in the supernatant organic phase at 0 °C after reaction, the same amount of acetophenone and small quantity of $(CH_3)_3COK$ were added to perform the next run. This procedure was repeated six times and the results indicated that the catalyst could be reused at least six times without much loss of the catalytic activity (Fig. 3). According to the ICP-AES characterization of the spent catalyst, the leaching of Ru species from the catalyst was only around 1.83 wt% after six runs, indicating that the

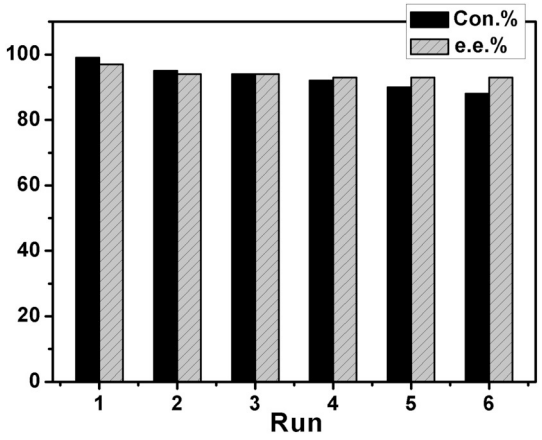


Fig. 3. Recyclability of catalyst [PEG-1000-C_{12}MIM][Ru-BINAP-DPENDS] for asymmetric hydrogenation of acetophenone.

catalyst was resistant to leaching during temperature-regulated phase separation and a clean separation has been obtained efficiently by this thermoregulated catalyst system. Besides, the FT-IR spectrum of the spent catalyst showed the preservation of the catalyst structure after reuses (Fig. 1d), which could account for the excellent recyclability of the catalysts upon recycling.

For the asymmetric hydrogenation reaction, as we have observed previously, the alkaline condition seemed to be very important (Table S1), which could accelerate the splitting of hydrogen to form H^- and H^+, and thus improve the activity of the reaction [27]. We then analyzed ^1H NMR spectra of the present ongoing catalytic system, which was very air-sensitive (Fig. 5Sc). A faint singlet at −7.2 ppm attributable to the Ru−H bond was observed in the presence of H_2 (Fig. 5Sb). This result supported the hydrogen transfer mechanism, and confirmed that the Ru−H bond was formed during the reaction [28]. With the help of the base promoter, hydrogen was likely split to form H^- and H^+, hereby Ru(II) and H^- formed a Ru−H bond. The Ru−H bond with adjacent N−H and C═O in the prochiral ketone formed a six-membered ring, and thereby a chiral alcohol was generated. In this mechanism, the most critical step was the formation of the Ru−H bond (Fig. 5S).

3. Conclusion

In summary, this work developed an attractive approach to design the thermoregulated IL-coordinating ruthenium complexes as new recyclable homogeneous catalysts. A variety of the aromatic ketones has been converted efficiently into corresponding chiral alcohols by the present thermoregulated catalysts using molecular hydrogen as hydrogen donor under mild conditions. Especially, the appropriate coupling of the IL cation and anion moiety was very vital, which offered not only excellent activity and enantioselectivity, but also a clean thermoregulated phase-separation. The activation of dihydrogen is proposed to involve its heterolytic splitting across the metal-hydrides, accelerated by base promoter. The present thermoregulated IL system may provide an effective approach for the separation of various homogeneous catalysts for the other organic reactions.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.catcom.2018.12.014.

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